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(71) Applicant: **UNILEVER PLC**
Unilever House Blackfriars P.O. Box 68
London EC4P 4BQ(GB)

(84) Designated Contracting States:
GB

(71) Applicant: **UNILEVER NV**
Burgemeester s' Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)

(84) Designated Contracting States:
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(72) Inventor: **Naik, Appaya Raghunath**
8 Kilmalcolm Close Wexford Road
Oxton Birkenhead Merseyside(GB)

(72) Inventor: **Coxon, Andrew Charles**
29 Gotham Road, Dibbens Hey
Bebington Wirral L63 9NG(GB)

(74) Representative: **Gura, Henry Alan et al,**
MEWBURN ELLIS & CO. 2/3 Cursitor Street
London EC4A 1BQ(GB)

(54) Detergent compositions.

(57) A liquid detergent composition suitable especially for dishwashing by hand contains four components:

- (a) any anionic detergent except alkyl ether sulphate
- (b) alkyl ether sulphate (any alkyl sulphate therein contributing to component (a) above)
- (c) a lather booster which is an alkanolamide, dialkanolamide, betaine or amine oxide
- (d) a non-ionic detergent, preferably an ethoxylated non-ionic of HLB value 12.0 to 16.0.

The amount of (a) is limited to an amount depending on its qualitative nature, and the ratio of (a) to (b) is 2:1 to 1:10.

Compositions of the invention can provide a combination of satisfactory performance, particularly good mildness and cost effectiveness.

DETERGENT COMPOSITIONS

5 The present invention relates to liquid detergent compositions suitable for use especially, but not exclusively, in fabric washing, shampoos, and above all, in manual dishwashing operations in both hard and soft water.

10 The term "dishes" as used herein means any utensils involved in food preparation or consumption which may be required to be washed to free them from food particles and other food residues, greases, proteins, starches, gums, dyes, oils and burnt organic residues.

15 Light-duty liquid detergent compositions such as are suitable for use in washing dishes are well-known. Many of the formulations in commercial use at the present time are based on a sulphonate-type anionic detergent, especially on alkyl benzene sulphonate, in conjunction with an alkyl polyethoxy sulphate (alkyl ether sulphate). The sulphonate-type detergent generally predominates.

20 The use of conventional dishwashing liquids based on alkyl benzene sulphonate/alkyl ether sulphate or on alkyl sulphate/alkyl ether sulphate is seen to have deleterious influence on hand conditions of users. Hence mildness in washing-up liquid is considered a desirable quality, and many specially formulated products on the market make claims for it.

25 Protein denaturation by surfactants is considered to be

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one of the major causes of skin irritation and skin roughness induced by surfactants (G. Imokawa et al JAOCS 52 484-489 Dec. 1975). The degree of surfactant denaturation of protein depends on the type of surfactants and their concentration.

5 At present the dishwashing formulations that are on the market which are less interactive with protein and hence considered to be milder are those based on a combination of ether sulphates and amine oxides. See for example US-A-3 928 249 (Procter and Gamble). In place of amine oxides, betaines can also
10 be used. See for example US 4 554 098. However, such formulations - particularly with amine oxides - are expensive not only because the active ingredients are expensive but also because they require a large amount of expensive hydrotropes such as xylenesulphonate and/or ethanol to produce liquids which are stable
15 and of acceptable viscosity. Therefore there is a need for the development of more cost-effective mild dishwashing formulations, which are based on relatively less expensive detergent raw materials and which also require lesser amounts of expensive hydrotropes.

20 The present invention is based on the realisation that cost-effective mild liquid dishwashing formulations with adequate performance can be obtained by careful choice of the active ingredients and their preparations in particular by restricting the main anionic detergent active present and including also selected
25 amounts of alkyl ether sulphate, lather boosters and non-ionic

detergent active material.

According to a first aspect of the present invention there is provided a stable detergent composition containing from 10 to 80% by weight of an active detergent mixture and also containing water, the active detergent mixture comprising:-

- (a) anionic detergent active other than alkyl ether sulphate;
- (b) alkyl ether sulphate containing one or more alkylene oxide residues per molecule, the weight ratio of components (a) and (b) being in the range 2:1 to 1:10, preferably in the range 2:1 or 1.5:1 to 1:3, more preferably 1.5:1 to 1:2. The proportion of component (b) with a single ethylene oxide residue per molecule being not substantially greater than the proportion with the commonest higher number of ethylene oxide residues per molecule;
- (c) a lather booster selected from monoalkanolamide, dialkanolamide, betaine, amine oxide and mixtures thereof, the total amount of said lather booster being in the range 5 to 30% by weight of the active detergent mixture, the amount of monoalkanolamide not exceeding 20% by weight, and the total of monoalkanolamide, dialkanolamide and betaine, plus $1\frac{1}{2}$ times the amount of amine oxide being at least 7.5% by weight of the active detergent mixture; and
- (d) nonionic detergent active material in an amount in the range 5 to 35% by weight of the active detergent mixture.

For reasons of cost-effectiveness, and availability of materials, at least a major proportion of the component (a) is

selected from secondary alkane sulphonate, alkylbenzene sulphonate, primary alkyl sulphate, fatty acyl ester sulphonate, dialkylsulphosuccinate and alpha-olefin sulphonate.

The anionic detergent active (a) must not exceed a maximum amount which is dependent on the nature of this active. For instance if secondary alkane sulphonate is substantially the only active (a) then the amount of it should not substantially exceed 50% by weight of the weight of the active detergent mixture.

Maxima for other detergent actives, when substantially alone, are:

| | |
|-----------------------------|-----|
| alkyl aryl sulphonate | 30% |
| alkyl sulphate | 40% |
| fatty acyl ester sulphonate | 50% |
| dialkylsulphosuccinate | 60% |
| alpha olefin sulphonate | 60% |

If anionic detergent active (a) is a mixture, then the maximum for the mixture is calculated as the total of the above maxima scaled in proportion to the amount of each active in the mixture.

Primary alkane sulphonate is preferably not used, but the maximum amount of it permitted in a mixture is calculated on the basis that the maximum amount of it, if substantially alone, is 40% by weight of the active detergent mixture.

The maximum amount of anionic detergent active (a) can

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conveniently be expressed by specifying that the active detergent mixture has maximum contents:

u of alkyl aryl sulphonate

v of primary alkane sulphonate

5

w of alkyl sulphate

x in total of secondary alkane sulphonate and

fatty acyl ester sulphonate

y in total of dialkylsulphosuccinate and alpha-olefin sulphonate, and

10

z in total of any other anionic active present - except of course for the alkyl ether sulphate which is component (b)-

where u, v, w, x, y and z are all percentages by weight of the detergent active mixture and are given by the relationship:

15

$$\frac{u}{30} + \frac{v}{40} + \frac{w}{40} + \frac{x}{50} + \frac{y}{60} + \frac{z}{60} \leq 1$$

Generally the anionic detergent active (a) is at least 10% by weight of the active detergent mixture, preferably at least 15% or 20%. It does not provide more than 60% of the active detergent mixture.

20

Preferred maxima for anionic detergent actives are lower:

| | |
|-----------------------|-------|
| alkyl aryl sulphonate | 22.5% |
|-----------------------|-------|

| | |
|----------------|-----|
| alkyl sulphate | 30% |
|----------------|-----|

| | |
|-----------------------------|-------|
| secondary alkane sulphonate | 37.5% |
|-----------------------------|-------|

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| | |
|-----------------------------|-------|
| fatty acyl ester sulphonate | 37.5% |
|-----------------------------|-------|

| | |
|------------------------------------|------------|
| dialkylsulphosuccinate | 50% |
| alpha-olefin sulphonate | 50% |
| primary alkane sulphonate | 30% |
| any other anionic detergent active | 50% or 40% |

5 Compositions of the invention can give performances in detergency tests as good as those of conventional compositions containing alkyl benzene sulphonate, but are considerably milder.

A significant form of this invention employs secondary alkane sulphonate as anionic detergent active (a) and
 10 monoethanolamide or diethanolamide as the lather booster (c), the active detergent mixture having in addition to the secondary alkane sulphonate and alkyl sulphate introduced with the alkyl ether sulphate a maximum content of 5% in total of alkyl sulphate, primary alkane sulphonate and alkylaryl sulphonate
 15 material and preferably being substantially free of such additional sulphate and sulphonate material.

It is particularly surprising that the harsh actives alkylarylsulphonate and primary alkyl sulphate can be tolerated at levels as high as 30 and 40% by weight of the detergent active
 20 mixture. A significant aspect of this invention therefore resides in a four component composition as specified above in which primary alkyl sulphate, alkylbenzene sulphonate or a mixture of the two constitutes at least 10% of the active detergent mixture and at least a majority of the component (a).

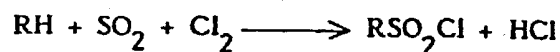
25 In this invention it may be preferred that the total

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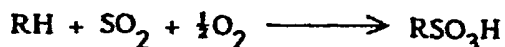
amount of alkyl benzene sulphonate, primary alkyl sulphate and primary alkane sulphonate are no greater than the total of components (c) and (d). The total of these anionic detergents plus any secondary alkane sulphonate and fatty acyl ester sulphonate may be no greater than the total of (c) and (d). The total amount of component (a) is preferably no greater than $1\frac{1}{4}$ times the total of (c) and (d). In some forms of the invention the total amount of (a) is no greater than the total of (c) and (d).

Although in principle the concentration of the active detergent mixture may be as high as desired in the range 10 to 80%, provided that a stable liquid or gel product can be obtained, the range of 5 to 60% by weight is preferred, and aqueous liquid compositions with an active detergent mixture in the range of 5 to 40% by weight are of especial interest.

Six detergent actives have been mentioned above as possibilities for component (a). Of these, secondary alkane sulphonate may be preferred. It is generally produced by a free radical reaction, either a sulphochlorination reaction (Reed reaction) of a paraffin



followed by hydrolysis and neutralisation, or by a sulphoxidation reaction



followed by neutralisation.

The secondary alkane sulphonate component (a) will normally be a mixture of materials of different alkyl chain lengths, of the formula:



where R_1 and R_2 which may be the same or different are each a straight or branched chain alkyl group having at least one carbon atom, the alkyl chain length (i.e. total number of carbon atoms of R_1 and R_2 plus 1) preferably being in the range 13 to 18, and X is a solubilising cation. An example of such material is SAS 60 of Hoechst (SAS is a Trade Mark) which is produced by a sulfoxidation process.

A suitable secondary alkane sulphonate produced by a sulphochlorination process is Mersolat (trade mark) of Bayer.

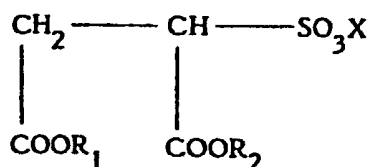
Alkylbenzene sulphonate for use in component (a) is preferably derived from an alkylbenzene with a C_8 to C_{16} primary or secondary alkyl group. This may in particular be C_8 to C_{13} alkyl. Suitable alkylbenzene sulphonates are Dobs 102 of Shell and Marlon A of Hlls.

Primary alkyl sulphate (primary alcohol sulphate) for use in component (a) is preferably of the formula



5 where R is a C₈ to C₁₆ primary alkyl group and X is a solubilising cation. Suitable is Dobanol 23A of Shell in which R is predominantly C₁₂ and C₁₃. As will be explained in more detail below alkyl sulphate is a constituent of alkyl ether sulphates which provide component (b).

10 Suitable detergent-active dialkyl sulphosuccinates are compounds of the formula



15 wherein each of R₁ and R₂, which may be the same or different, represents a straight-chain or branched-chain alkyl group having 3 to 12 carbon atoms, preferably from 4 to 10 carbon atoms and more preferably from 6 to 8 carbon atoms, and X represents a
20 solubilising cation.

The alkyl groups R₁ and R₂ are preferably straight-chain or (in mixtures) predominantly straight-chain.

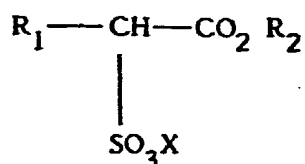
Among dialkyl sulphosuccinates that may advantageously be used in the composition of the invention are the C₆/C₈
25 unsymmetrical materials described and claimed in GB 2 105 325

(Unilever); the dioctyl sulphosuccinate/dihexyl sulphosuccinate mixtures described and claimed in GB 2 104 913 (Unilever); and the mixtures of symmetrical and unsymmetrical dialkyl sulphosuccinates described and claimed in GB 2 108 520 (Unilever).

Appropriate alpha-olefin sulphonates will generally be in the range having from twelve to sixteen carbon atoms.

An example of such a material is Liporan 440, a C₁₄ alpha-olefin sulphonate from Lion Corporation, Japan

Appropriate fatty acid ester sulphonates are of formula



where R₁ is straight or branched C₈ to C₁₆ alkyl

R₂ is straight or branched C₁ to C₄ alkyl

and X is a solubilising cation.

R₁ is preferably C₁₀ to C₁₂

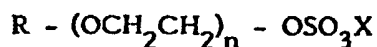
and R₂ is preferably butyl.

Component (a) of the active detergent mixture may also include one or more other detergent actives used in liquid compositions, for example alkyl glyceryl ether sulphonates, or alkyl sarcosinates. The amount is preferably less than half of component (a), more preferably not more than a quarter of it.

The second essential component (b) of the active

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detergent mixture is alkyl ether sulphate (sometimes called alcohol ether sulphate or alkyl polyethoxy sulphate) having at least one ethylene oxide residue per molecule. This will normally be provided by incorporating into the composition an alkyl ether sulphate which is a mixture of materials of the general formula:



wherein R is a C₁₀ to C₁₈ primary or secondary alkyl group, X is a solubilising cation, and n, the average degree of ethoxylation, is at least 1.5, preferably from 2 to 12, more preferably 3 to 8.

Particularly preferred values of n are 3, 4 and 5. R₃ is preferably a C₁₀ to C₁₆ alkyl group. In any given alkyl ether sulphate, a range of differently ethoxylated materials, and some unethoxylated material, will be present and the value of n represents an average. The unethoxylated material is, of course, alkyl sulphate and this contributes to component (a).

The amount of alkyl sulphate in any alkyl ether sulphate will depend on average degree of ethoxylation n. When n is 3, alkyl sulphate typically constitutes 15 to 20% of the mixture, and less than this when n is 4 or more. When the proportion of alkyl sulphate is low, it may prove convenient to ignore it. Nevertheless it contributes to component (a).

When the average degree of ethoxylation is 2, alkyl sulphate typically constitutes 30% of the mixture provided as

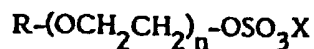
"alkyl ether sulphate". Such a mixture can provide both component (b) and component (a), with the latter then consisting entirely of alkyl sulphate.

5 We have found that it is not feasible to use alkyl ether sulphate with an average degree of ethoxylation below 1.5. Unless the alkyl sulphate content of the ether sulphate is providing much or the whole of component (a), it is preferred that the alkyl ether sulphate is provided by material with an average of at least 2 or 2.5 ethylene oxide residues per molecule.

10 An alkyl ether sulphate contains molecules with differing numbers of ethylene oxide residues in a statistical distribution. In an alkyl ether sulphate where the average degree of ethoxylation is 1.5 or greater, the proportion of molecules with a single ethylene oxide residue will not be substantially greater than the
15 proportion with two ethylene oxide residues, nor the proportion with the most frequently encountered number of ethylene oxide residues (if this is more than two). Therefore this feature is an observable characteristic of component (b) and of compositions of this invention. If the average degree of ethoxylation is 2 or more,
20 as preferred, the proportion of molecules with a single ethylene oxide residue will be less than the proportion with two ethylene oxide residues and the proportion with the most frequently encountered number.

25 Preferred alkyl ether sulphates of the component (b), excluding alkyl sulphate, are mixtures of compounds of the above

formula



5 in which n is any positive integer.

It is preferred, for this invention, to use primary alkyl ether sulphates containing less than 20% by weight of material with alkyl groups of C₁₄ and above material, more preferably substantially C₁₂ and C₁₃ material only. Such material preferably
10 has an average degree of ethoxylation of 2 to 6, more preferably 3 to 5.

Examples of preferred alkyl ether sulphates for use in the present invention are Dobanol (Trade Mark) 23-3 from Shell in which the degree of ethoxylation (n) is 3 and the equivalent
15 material in which the degree of ethoxylation is 4. These materials which are based on C₁₂-C₁₃ (50% of each) primary alcohol (about 75% straight chain, 25% 2-methyl branched). Another preferred material is an alkyl ether sulphate based on Lial (Trade Mark) 123 from Chimica Augusta, which is a branched chain primary alcohol
20 with a degree of ethoxylation of 3 to 4.

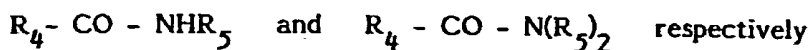
A suitable example of a secondary alcohol ether sulphate is a material derived from an alcohol such as Tergitol 15/S/3 (trade mark) of Union Carbide (this material itself is not at present available). The conventional process of manufacture of
25 secondary alkyl ether sulphates is such that there is only a very

small quantity of alkyl sulphate in the product.

Component (b) provides at least 12% of the active detergent mixture, preferably it provides at least 15 or 20%. It preferably provides not more than 60 or 65% of the active detergent mixture.

5 The solubilising cations of the anionic detergent actives of components (a) and (b), denoted as X in formulae above, may be any which provide the desired solubility of the anionic material. Monovalent cations such as alkali metal ions, ammonium and substituted ammonium are typical. Divalent ions giving adequate solubility may be used, and
10 especially magnesium ions may be present to improve soft water performance and can be incorporated as magnesium salt of the anionic actives or as inorganic magnesium salts, or in the hydrotrope system.

Component (c) of the active detergent mixture of the composition of the invention is preferably one or more mono- or
15 dialkanolamides, preferably C_8 to C_{18} , more preferably C_{10} - C_{18} carboxylic acid mono- or di(C_2 - C_3) alkanolamides. These have the general formulae



wherein R_4 is a C_7 - C_{17} aliphatic group, preferably straight-chain and
20 preferably saturated, and R_5 is a hydroxyethyl or hydroxypropyl group. R_5 is preferably a 2-hydroxyethyl group.

Materials of this type are generally made from fatty

acids of natural origin and contain a range of molecules having R_4 groups of different chain lengths; for example, coconut ethanolamides consist predominantly of C_{12} and C_{14} material, with varying amounts of C_8 , C_{10} and C_{16} material. Preferred are ethanolamides derived from
5 so-called middle cut coconut fatty acid, most preferably from lauric acid.

Monoalkanolamide may be used alone (in an amount in the range 7.5 to 20% of the active detergent mixture) or dialkanolamide may be used alone (in an amount in the range 7.5 to 30% of the active detergent mixture). It is preferred to use a mixture of mono- and
10 dialkanolamide, most preferably in a weight ratio of 2:1 to 1:2.

The other possibilities for component (c) are betaines and amine oxides. It is preferred to avoid using substantial amounts of these, especially amine oxides, for the sake of economy and consequent cost effectiveness. Preferably then the amount of amine oxide is not
15 more than 10% by weight of the active detergent mixture. Preferably the amount of betaine is not more than 20% by weight of this mixture. The total amount of amine oxide and betaine is preferably not more than 20% and more preferably not more than 15% or 10% by weight of the active detergent mixture.

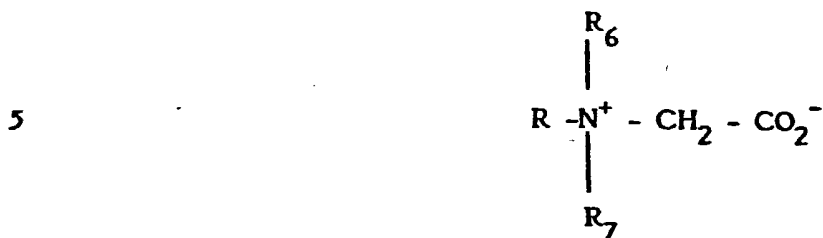
20 It may be convenient to use amine oxide and/or betaine at such low levels, as part or even all of component (c).

In important forms of the invention, however, amine oxide

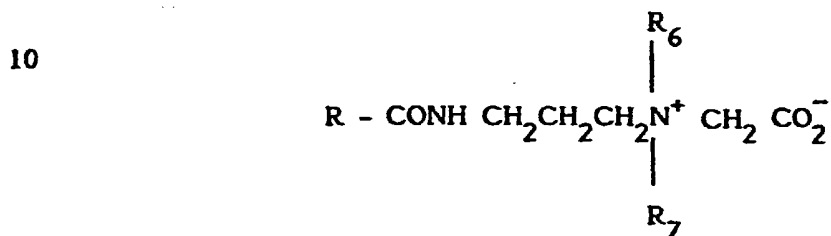
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is excluded.

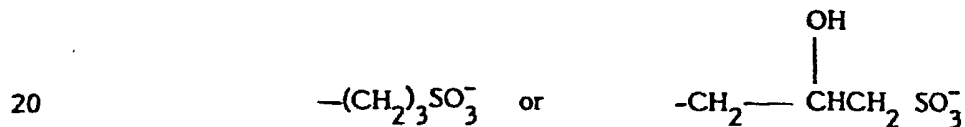
Suitable betaines include simple betaines of formula



and amido betaines of formula:

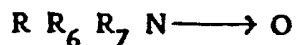


15 In both formulae R is a C₇ to C₁₆ straight or branched alkyl group. It may be a lauryl group or a middle cut coconut alkyl group. R₆ and R₇ are each C₁ to C₃ alkyl or C₁ to C₃ hydroxyalkyl. Examples of sulphobetaines have the above formulae with -CH₂CO₂⁻ replaced by



A suitable simple betaine is Empigen BB from Albright and Wilson. It has the formula quoted above in which R is C₁₂ to C₁₄ alkyl, derived from coconut, and R₆ and R₇ are both methyl.

Suitable amine oxides have the formula



wherein R is a straight or branched chain C₈ to C₁₈ alkyl group
 5 and R₆ and R₇ are each C₁ to C₃ alkyl, or C₁ to C₃
 hydroxyalkyl. A suitable amine oxide is Empigen OB from Albright
 and Wilson. In it R is C₁₂ to C₁₄ alkyl, derived from coconut
 and R₆ and R₇ are both methyl.

Component (c) preferably constitutes at least 10% e.g.
 10 12% to 25%, by weight of the active detergent mixture.

Component (d) is one or more non-ionic detergent active
 materials, e.g. materials conventionally used in detergent
 formulations. The alkanolamides, betaines and amine oxides of
 component (c) do not form part of component (d).

15 Component (d) is preferably a polyalkoxylated material,
 notably it is one or more ethoxylated non-ionic detergent active
 materials. It is then desirable that such material should have an
 HLB value in the range from 12.0 to 16.0.

Component (d) may be a polyethoxylated aliphatic alcohol
 20 having an alkyl chain length of from C₈ to C₁₈ preferably C₈ to
 C₁₆, and an average degree of ethoxylation of from 4 to 14.
 Suitable nonionic detergents include short-chain high-foaming
 ethoxylated alcohols of the general formula



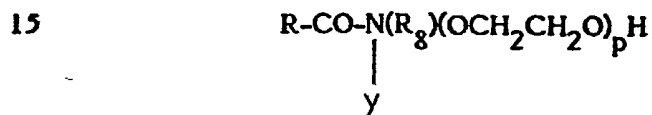
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wherein R is an alkyl group, preferably straight-chain, having from 8 to 18 better 8 to 16 and yet more preferably 9 to 11, carbon atoms, and the average degree of ethoxylation \underline{m} is from 5 to 14, more preferably 6 to 12. An especially preferred nonionic detergent is Dobanol 91-8 from
 5 Shell, in which R is C_9-C_{11} (predominantly straight-chain) and \underline{m} is 8.

Alternative suitable materials are those in which R is a secondary alkyl having from 8 to 18, preferably 11-15, carbon atoms and \underline{m} is from 5 to 14 preferably 6-12. An example is Tergitol 15/5/12 of Union Carbide (not available at present) or the material of the Softanol
 10 A series (from Japan Catalytic).

Preferably the polyethoxylated alcohol mixture is stripped, to reduce odour imparted to the composition.

Another possibility for the component (d) is an ethoxylated alkanolamide of the general formula



wherein R is a straight or branched alkyl having from 7 to 18 carbon atoms,

20 R_g is an ethyleneoxy or propyleneoxy group

Y is hydrogen or $-R_g(\text{CH}_2\text{CH}_2\text{O})_q\text{H}$

p is 1 or more and q is 0, 1 or more.

R may be lauryl or coconut alkyl. Examples of

ethoxylated alkanolamide are Amidox L5 and Amidox C5 from Stepan Chemical Company.

Further possibilities for component (d) are ethoxylated alkylphenols and ethoxylated fatty acids, i.e. polyethyleneglycol esters of fatty acids.

Component (d) preferably constitutes at least 10% by weight of the active detergent mixture e.g. 12 to 25%. The total of components (c) and (d) together may constitute at least 25% by weight of the active detergent mixture.

As well as the active detergent mixture and water, the liquid detergent compositions of the invention will generally need to contain one or more hydrotropes. Hydrotropes are materials present in a formulation to control solubility, viscosity, clarity and stability but which themselves make no active contribution to the performance of the product. Examples of hydrotropes include lower aliphatic alcohols, especially ethanol; urea; lower alkylbenzene sulphonates such as sodium, toluene and xylene sulphonates and combinations of these. Preferred are alcohol, urea, and xylene sulphonate. Hydrotropes are expensive and take up room in a formulation without contributing to its performance, and it is therefore desirable to use as small quantities of them as possible.

For example, the use of amine oxides as mentioned above requires a large amount of alcohol as hydrotrope. For this reason and because of expense, it is preferred to avoid the use of a

substantial amount of any tertiary amine oxide in the present invention.

5 In preferred forms of this invention the weight of hydrotrope in the composition is not more than 12% of the weight of the active detergent mixture.

The compositions of the invention may also contain the usual minor ingredients such as perfume, colour, preservatives and germicides.

10 The stable liquid detergent compositions of the invention may be used for all normal detergent purposes especially where foaming is advantageous, for example, fabric washing products, general purpose domestic and industrial cleaning compositions, carpet shampoos, car wash products, personal washing products, shampoos, foam bath products, and above all, manual dishwashing.

15 The invention is further illustrated by the following non-limiting Examples.

EXAMPLE 1

20 The foaming performances of various aqueous formulations were compared using a plate washing test. In the test, plates soiled with a standard starch/fat/fatty acid mixture were washed in a standard manner with 5 litres of test solution (total concentration of the product 1 g/litre in 24⁰H (French hardness) water at 45⁰C in a bowl, until a third of the surface of the solution in the bowl was covered with foam. The number of
25 plates washed before this arbitrary end-point was reached was

taken as an indicator of dishwashing and foaming performance.

In Table 1 below, the content of each component is given as percentage by weight of the whole composition. For each composition the total active detergent concentration (including the ethanolamide materials) was kept constant at 40%.

In Table 2 below the content of the same compositions is expressed as percentages by weight of the detergent active mixture, with the primary alkyl sulphate (from the alkyl ether sulphate) shown separately.

The foaming performance, cloud point and viscosity of various compositions of the invention (I-VI), a comparable composition containing alkyl benzene sulphonate (A), a comparable composition containing only secondary alkane sulphonate and alkyl ether sulphate (B), and a comparable composition containing no sulphonate (C) were obtained.

The lauric diethanolamide used was a commercial narrow-cut coconut diethanolamide, referred to as LDEA, and the cocomonoethanolamide was Empilan CME of Albright & Wilson.

The formulations and results are set out in Table 1 below and repeated in Table 2 expressed as percentages by weight of the active detergent mixtures.

TABLE I

| Composition No. | I | II | III | IV | V | VI | A | B | C |
|--------------------------------------|---|------------------------------|--------------------------------------|-----|-----|-----|-----|-------------------|-----|
| <u>Component</u> | <u>Percentages by weight of the formulation</u> | | | | | | | | |
| SAS60 | 12 | 12 | 12 | 13 | 13 | 13 | - | 32 | - |
| Marlon A | - | - | - | - | - | - | 12 | - | - |
| Dobanol 23-3A | 18 | 18 | - | 13 | - | 13 | 18 | 8 | 24 |
| Dobanol 23-4S | - | - | 18 | - | 13 | - | - | - | - |
| CEA | 4 | 4 | 4 | 4 | 4 | - | - | - | 4 |
| LDEA | - | - | - | 4 | 4 | 8 | 4 | - | 4 |
| Dobanol 91-8EO | 6 | 6 | 6 | 6 | 6 | 6 | 6 | - | 8 |
| Urea | 4 | - | 4 | 4 | 4 | 4 | 5 | 3 | - |
| IMS | - | 3 | - | - | - | - | 4 | 3 | 9 |
| MgSO ₄ ·7H ₂ O | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | - | 2 |
| Cloud pt. (°C) | +2 | -5 | +1 | -4 | -4 | -4 | -4 | -1 | +13 |
| Viscosity (mPa.s) | 208 | 168 | 320 | 224 | 320 | 272 | 196 | 280 | 192 |
| Plates Test | 27 | 27 | 28 | 28 | 28 | 26 | 30 | 27 | 21 |
| SAS* 60 | - | C ₁₃₋₁₈ | secondary alkane sulphonate | | | | - | Hoechst | |
| Marlon* A | - | C ₁₀₋₁₃ | alkyl benzene sulphonate | | | | - | HÜls | |
| Dobanol* 23-3A | - | C ₁₂₋₁₃ | alcohol 3EO(ethylene oxide) sulphate | | | | - | Shell | |
| Dobanol* 23-4S | - | C ₁₂₋₁₃ | alcohol 4EO sulphate | | | | - | Shell | |
| CEA | - | coco monoethanolamide | (Emplian* CME) | | | | - | Albright & Wilson | |
| LDEA | - | lauric diethanolamide | | | | | - | Shell | |
| Dobanol* 91-8EO | - | C ₉₋₁₁ | alcohol 8EO | | | | - | Shell | |
| IMS | - | industrial methylated spirit | | | | | - | Shell | |
| * | - | trade mark | | | | | - | Shell | |

The compositions (I-VI) of the invention had acceptable hydrotrope levels and viscosities and foaming performances only slightly inferior to composition A, as good as that of composition B and much better than that of composition C.

5 Mildness of some of the above formulations was estimated using a test of protein denaturation potency.

Several in vitro and in vivo methods for evaluating protein denaturation potency of surfactants and their mixtures have been reported (see K. Miyazawa et al Int J Cos Sci 6 33-46 1984
10 and the references cited therein). One such method is the study of interaction of skin acid phosphatase enzyme with detergents under realistic conditions reported by Prottey et al (Int J Cos Sci 6 263-273 1984).

Formulations II, IV, A, B, and C above were tested by
15 Prottey's method using dilute solutions at active concentrations giving equal plates test performance. The results expressed qualitatively as a rank order of predicted mildness are as follows:

$$C > IV \approx II > A \approx B$$

C was significantly better than IV and II, which were close
20 together. A and B gave results similar to each other and very much worse than IV and II.

EXAMPLE 2

Human volunteers were employed to compare the active detergent mixtures of compositions II, IV and V and a commercial
25 product identified as relatively mild. The detergent active in this

product was a 4:1 mixture of secondary alkane sulphonate and alkyl ether sulphate (average 3 ethylene oxide residues per molecule). Thus it was closely similar to composition B.

The compositions were prepared at an active detergent concentration of 25% by weight without hydrotropes. Each trial involved panellists washing each volar forearm with a different one of the above products 4 times daily for 4 days and 3 times on day 5. For each wash 0.5 g of product was dosed onto the wetted volar forearm and it was washed with a gloved hand for 60 seconds. The forearm was rinsed after each wash. The products were randomly allocated to the panellists. Each volar forearm was graded for erythema dryness before each wash and 2 hours after the final wash. Any site reaching an erythema score denoting moderate erythema or a dryness score denoting moderate to severe dryness was discontinued.

The results showed that for potential to induce irritation V was significantly better than II and IV which were significantly better than the commercial product (composition B). For potential to induce skin dryness V was better than II or IV which were significantly better than composition B. These results are consistent with Example 1.

EXAMPLE 3

A number of formulations were tested for interaction with skin acid phosphatase, by Prottey's method, using solutions of equal dilution. The compositions and the percentage inhibition (i.e.

26.

100% minus percentage activity remaining) are set out in Table 3 below, where the compositions are expressed as percentages by weight of the formulation and then repeated in terms of percentages by weight of active detergent mixtures. Water gave no inhibition at all, i.e. 100% of activity remained.

5

TABLE 3

| Composition no: | 3.1 | 3.2 | 3.3 | 3.4 | 3.5 | 3.6 | 3.7 | 3.8 | 3.9 | 3.10 | 3.11 |
|---|--|-----|-----|------|------|------|------|-----|--------|------|------|
| Component | Percentages by weight of the formulation | | | | | | | | | | |
| SAS60 | 20 | 16 | 16 | 12 | 6 | 12 | 12 | 16 | 12 | 18 | 12 |
| Dobs 102 | - | - | - | 6 | - | - | - | - | - | - | - |
| Dobanol 23A | - | - | - | - | 12 | - | 6 | - | - | - | - |
| Dobanol 23-4A | 20 | 16 | 16 | 10 | 10 | 14 | 10 | 16 | - | 10 | 12 |
| Dobanol 23-2.5A | - | - | - | - | - | - | - | - | 12 | - | - |
| CEA | - | 4 | - | 4 | 4 | - | 4 | - | 4 | 4 | 4 |
| LDEA | - | 4 | - | - | - | 12 | - | - | 4 | - | 4 |
| Dobanol 91-8EO | - | - | - | 8 | 8 | 2 | 8 | 8 | 8 | 8 | 8 |
| Percentages by weight of active detergent mixture | | | | | | | | | | | |
| SAS60 | 50 | 40 | 40 | 30 | 15 | 30 | 30 | 40 | 30 | 45 | 30 |
| Dobs 102 | - | - | - | 15 | - | - | - | - | - | - | - |
| alkyl sulphate | 5 | 4 | 4 | 2.5 | 32.5 | 3.5 | 17.5 | 4 | 5.5 | 2.5 | 3 |
| alkyl ether sul- phate excluding) alkyl sulphate) | 45 | 36 | 36 | 22.5 | 22.5 | 31.5 | 22.5 | 36 | 24.5 | 22.5 | 27 |
| CEA | - | 10 | - | 10 | 10 | - | 10 | - | 10 | 10 | 10 |
| LDEA | - | 10 | - | - | - | 30 | - | - | 10 | - | 10 |
| Dobanol 91-8EO | - | - | - | 20 | 20 | 5 | 20 | 20 | 20 | 20 | 20 |
| % Inhibition | 72 | 63 | 61 | 58 | 44 | 41 | 41 | 41 | 23 | 21 | 13 |
| Dobs 102 | C ₁₀ -C ₁₂ alkylbenzene sulphonate | | | | | | | | -Shell | | |
| Dobanol 23A | C ₁₂ -C ₁₃ primary alkyl sulphate | | | | | | | | -Shell | | |
| Dobanol 23-4A | C ₁₂ -C ₁₃ alcohol 4EO (ethyleneoxide)sulphate | | | | | | | | -Shell | | |
| Dobanol 23-2.5A | C ₁₂ -C ₁₃ alcohol 2.5EO sulphate | | | | | | | | -Shell | | |

EXAMPLE 4

In this and subsequent examples mildness is assessed by measuring the inhibition of wheatgerm acid phosphatase (WGAP). This has been found to correlate well with the results obtained with human volunteers: for instance the inhibition of WGAP by compositions B, IV and V in a series of comparative experiments was 100%, 75% and 69% respectively.

It is notable that the commercial product (composition B) gave total inhibition of this enzyme. Even a 3:7 mixture of SAS 60: Dobanol 23-3A gave 91% inhibition.

A series of compositions was assessed in which the nature of component (a) was varied, and the proportions of components (a) and (b) were also varied. These compositions were also assessed for foaming performance by means of a modified Schlachter-Dierkes test based on the principle described in Fette und Seifen 1951, 53, 207. A 100ml aqueous solution of each material tested, having a concentration of 0.04% active detergent in 24⁰H water (French hardness) at 45⁰C was rapidly oscillated using a vertically oscillating perforated disc within a graduated cylinder. After the initial generation of foam, increments (0.2g) of soil (9.5 parts commercial cooking fat, 0.25 parts oleic acid, 0.25 parts stearic acid and 10 parts wheat starch in 120 parts water) were added at 15-second intervals (10 seconds' mild agitation and 5 seconds' rest) until the foam collapsed. The result was recorded as the number

of soil increments (NSI score): a score difference of 6 or less is generally regarded as insignificant. Each result was typically the average of 3 or 4 runs.

5 The compositions and the results obtained are set out in Table 4 below. In this table the compositions are quoted, and then calculated figures are given for the amounts of alkyl sulphate and alkyl ether sulphate excluding alkyl sulphate.

10 As can be seen from Table 4, the compositions' foaming performance was satisfactory in that it was never worse than the control composition, effectively composition V, shown in the final column of Table 4. Inhibition was never the total inhibition encountered with composition B (i.e. the commercial product). It can be seen that levels of 25% alkyl benzene sulphonate with 3.5% alkyl sulphate, and a level of 37.5% alkyl sulphate give about 85%
15 inhibition, which is intermediate between the commercial product (composition B) and composition V.

TABLE 4

| Composition no: Component | 4.1 | 4.2 | 4.3 | 4.4 | 4.5 | 4.6 | 4.7 | 4.8 | 4.9 | 4.10 | 4.11 | 4.12 | 4.13 | V |
|---|---|------|-----|------|-----|------|-----|------|-----|------|------|------|------|------|
| Percentages by weight of active detergent mixture | | | | | | | | | | | | | | |
| SAS 60 | 20 | 25 | 30 | 35 | | | | | | | | | | 32.5 |
| Dobs 102 | | | | | 20 | 25 | 30 | 35 | | | | | | |
| Dobanol 23A | | | | | | | | | 20 | 25 | 30 | 35 | | |
| Dobanol 23-2A | | | | | | | | | | | | | 60 | |
| Dobanol 23-4A | 40 | 35 | 30 | 25 | 40 | 35 | 30 | 25 | 40 | 35 | 30 | 25 | | 32.5 |
| LME | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| LDEA | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 10 |
| Dobanol 91-8EO | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 | 15 |
| alkyl sulphate | 4 | 3.5 | 3 | 2.5 | 4 | 3.5 | 3 | 2.5 | 24 | 28.5 | 33 | 37.5 | 18 | 3 |
| alkyl ether sul- phate excluding) alkyl sulphate) | 36 | 31.5 | 27 | 22.5 | 36 | 31.5 | 27 | 22.5 | 36 | 31.5 | 27 | 22.5 | 42 | 29 |
| % Inhibition of WGAP | 54 | 64 | 68 | 75 | 74 | 83 | 89 | 95 | 58 | 69 | 77 | 82 | 65 | 72 |
| Foaming performance | 35 | 36 | 38 | 38 | 37 | 40 | 44 | 43 | 37 | 39 | 43 | 40 | 35 | 34 |
| Dobanol 23-2A LME | C ₁₂₋₁₃ alcohol 2EO (ethyleneoxide)sulphate lauric monothanolamide -Shell | | | | | | | | | | | | | |

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EXAMPLE 5

A number of compositions were assessed for WGAP inhibition. These included a series which were each the same as composition V, except that the secondary alkane sulphonate was replaced with an equal weight of another anionic detergent active. Some were also assessed for foaming performance by means of the modified Schlacter-Dierkes test referred to above. The compositions and the results obtained are set out in Table 5 below.

"Sulphosuccinate" denotes a statistical mixture (mole ratio 1:2:1) of di-n-octyl sulphosuccinate, n-hexyl n-octyl sulphosuccinate and di-n-hexyl sulphosuccinate (sodium salts), prepared from a 1:1 mixture of n-hexanol and n-octanol by the method described in Example 1 of GB 2 108 520 (Unilever).

"Butyl FAES" denotes the sulphonate of n-butyl laurate.

TABLE 5

| Composition no: V | 5.1 | 5.2 | 5.3 | 5.4 | 5.5 | 5.6 | 5.7 | 5.8 |
|--|--|------|------|------|------|------|------|-------|
| Component | <u>Percentages by weight of active detergent mixture</u> | | | | | | | |
| SAS 60 | 32.5 | | | | | | | |
| Dobs 102 | 32.5 | | | | | | | |
| Dobanol 23A | | 32.5 | | | | | | |
| Butyl FAES | | | 32.5 | | | | | |
| Sulphosuccinate | | | | 32.5 | | | 57 | 57 |
| C ₁₄ alpha-olefin sulphonate | | | | | 32.5 | | | |
| Dobanol 23-3A | | | | | | 28.5 | | |
| Dobanol 23-4A | 32.5 | 32.5 | 32.5 | 32.5 | 32.5 | 7.5 | | 28.5 |
| CEA | 10 | 10 | 10 | 10 | 10 | 10 | | |
| LDEA | 10 | 10 | 10 | 10 | 10 | 10 | 14.5 | 14.5 |
| Dobanol 91-8EO | 15 | 15 | 15 | 15 | 15 | 15 | | |
| % Inhibition of WGAP | 72 | 90 | 80 | 70 | 60 | 55 | 35 | 82 80 |
| Foaming performance | 34 | | | | 46.5 | | | 49 51 |

The mildness arising with the different actives is apparent. It can also be seen that the composition of this invention, with sulphosuccinate as component (a) was superior in mildness to the sulphosuccinate compositions which were 4:2:1 sulphosuccinate: alkyl ether sulphate: diethanolamide, yet was fairly close in foaming performance, and better than the composition with secondary alkane sulphonate (composition V of Example 1).

EXAMPLE 6

The foaming performance of some further compositions was assessed by means of the modified Schlacter-Dierkes test. The compositions and the results obtained are set out in Table 6 below.

TABLE 6

| <u>Component</u> | | <u>Percentages by weight of</u> <u>active detergent mixture</u> | | | | |
|------------------|------------------------------|--|------|------|------|------|
| | SAS 60 | 32.5 | 32.5 | | | 32.5 |
| 5 | Dobanol 23-A | | | 21 | 21 | 32.5 |
| | Dobanol 23-4A | 32.5 | 32.5 | 42 | 42 | |
| | Lauryl mono- ethanolamide | 10 | 10 | 10.5 | 10.5 | 10 |
| | LDEA | 10 | 10 | | | 10 |
| 10 | Empigen OB | | | 10.5 | | |
| | Empigen BB | | | | 10.5 | |
| | Dobanol 91-8EO | | | 16 | 16 | 15 |
| | Amidox L5 | 15 | | | | |
| 15 | Amidox C5 | | 15 | | | |
| Performance | | 41 | 37 | 45 | 50 | 36 |

Amidox L5 is lauric monoethanolamide 5EO (ethylene oxide)

Amidox C5 is coconut monoethanolamide 5EO

20 Both are supplied by Stepan Chemical Company.

Empigen OB is C₁₂ to C₁₄ alkyl dimethyl amine oxide.

Empigen BB is C₁₂ to C₁₄ alkyl dimethyl betaine.

CLAIMS:

1. A stable detergent composition in liquid or gel form containing from 10 to 80% by weight of active detergent mixture and water, the active detergent mixture comprising four
5 components as follows:

(a) an anionic detergent active other than alkyl ether sulphate;

(b) alkyl ether sulphate containing one or more ethylene oxide residues per molecule, the weight ratio of components (a) and (b) being in the range 2:1 to 1:10, the proportion of
10 component (b) with a single ethylene oxide residue per molecule being not substantially greater than the proportion with the commonest higher number of ethylene oxide residues per molecule;

(c) lather booster selected from monoalkanolamide, dialkanolamide, betaine, amine oxide and mixtures thereof in a
15 total amount in the range 5 to 30% by weight of the active detergent mixture, the amount of monoalkanolamide not exceeding 20% by weight, the amount of amine oxide not exceeding 10% by weight, and the total amount of monoalkanolamide, dialkanolamide, and betaine plus $1\frac{1}{2}$ times the amount of amine oxide being at
20 least 7.5% by weight of the active detergent mixture,

(d) nonionic detergent active material in an amount in the range 5 to 35% by weight of the active detergent mixture;

the active detergent mixture having maximum contents u
25 of alkylaryl sulphonate, v of primary alkane sulphonate, w of alkyl

5 sulphate, a maximum total content x of secondary alkane
sulphonate and fatty acyl ester sulphonate, a maximum total
content y of dialkylsulphosuccinate and alpha-olefin sulphonate and
a maximum total content z of any other anionic detergent active
except said alkyl ether sulphate,

where u, v, w, x, y and z are all percentages by weight
of the detergent active mixture and are given by

$$\frac{u}{30} + \frac{v}{40} + \frac{w}{40} + \frac{x}{50} + \frac{y}{60} + \frac{z}{60} \leq 1$$

10

2. A composition according to claim 1 wherein at least a
major proportion of said component (a) is selected from secondary
alkane sulphonate, fatty acyl ester sulphonate,
dialkylsulphosuccinate, alpha-olefin sulphonate, primary alkyl
15 sulphate, alkylbenzene sulphonate and mixtures thereof.

3. A composition according to claim 1 or claim 2 wherein u,
v, w, x, y and z are given by

$$20 \quad \frac{u}{22.5} + \frac{v}{22.5} + \frac{w}{30} + \frac{x}{37.5} + \frac{y}{40} + \frac{z}{40} \leq 1$$

4. A composition according to any one of the preceding
claims wherein component (a) comprises secondary alkane
sulphonate, and component (c) consists of monoalkanolamide and/or
25 dialkanolamide,

the active detergent mixture having, in addition to the secondary alkane sulphonate (a) and alkyl sulphate from the alkyl ether sulphate which provides component (b), a maximum content of 5% in total of alkyl sulphate, alkane sulphonate and alkylaryl sulphonate material.

5. A composition according to any one of the preceding claims wherein the secondary alkane sulphonate is a mixture of materials of different alkyl chain lengths, of the formula



where R_1 and R_2 are each a straight or branched chain alkyl group having at least one carbon atom, the alkyl chain length (i.e. total number of carbon atoms of R_1 and R_2 plus 1) being in the range 13 to 18, and X is a solubilising cation.

6. A stable detergent composition in liquid or gel form containing from 10 to 80% by weight of active detergent mixture and water, the active detergent mixture comprising four components as follows:

(a) an anionic detergent active other than alkyl ether sulphate; wherein a major fraction of component (a) is selected from primary alkyl sulphate, alkyl benzene sulphate or a mixture thereof, which primary alkyl sulphate, alkyl benzene sulphonate or

mixture thereof is at least 10% of the active detergent mixture;

(b) alkyl ether sulphate containing one or more ethylene oxide residues per molecule, the weight ratio of components (a) and (b) being in the range 2:1 to 1:10, the proportion of component (b) with a single ethylene oxide residue per molecule being not substantially greater than the proportion with the commonest higher number of ethylene oxide residues per molecule;

(c) lather booster selected from monoalkanolamide, dialkanolamide, betaine, amine oxide and mixtures thereof in a total amount in the range 5 to 30% by weight of the active detergent mixture, the amount of monoalkanolamide not exceeding 20% by weight, and the total amount of monoalkanolamide, dialkanolamide, and betaine plus $1\frac{1}{2}$ times the amount of amine oxide being at least 7.5% by weight of the active detergent mixture,

(d) nonionic detergent active material in an amount in the range 5 to 35% by weight of the active detergent mixture;

the active detergent mixture having maximum contents u of alkylaryl sulphonate, v of primary alkane sulphonate, w of alkyl sulphate, a maximum total content x of secondary alkane sulphonate and fatty acyl ester sulphonate, a maximum total content y of dialkylsulphosuccinate and alpha-olefin sulphosuccinate and a maximum total content z of any other anionic detergent active except said alkyl ether sulphate,

where u, v, w, x, y and z are all percentages by weight

of the detergent active mixture and are given by

$$\frac{u}{30} + \frac{v}{40} + \frac{w}{40} + \frac{x}{50} + \frac{y}{60} + \frac{z}{60} \leq 1$$

5 7. A composition according to any one of the preceding claims wherein the weight ratio of the components (a) and (b) is in the range 1.5:1 to 1:3.

10 8. A composition according to any one of the preceding claims wherein the total amount of alkyl aryl sulphonate, primary alkane sulphonate and alkyl sulphate is not substantially greater than the total amount of components (c) and (d).

15 9. A composition according to any one of the preceding claims wherein the amount of component (a) is not greater than $1\frac{1}{2}$ times the total amount of components (c) and (d).

20 10. A composition according to any one of the preceding claims wherein the total amount of amine oxide and betaine is not more than 20% by weight of the active detergent mixture.

25 11. A composition according to any one of the preceding claims wherein component (c) contains substantially no amine oxide.

40.

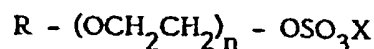
12. A composition according to any one of the preceding claims wherein component (d) constitutes at least 10% by weight of the active detergent mixture.

5 13. A composition according to any one of the preceding claims wherein each of components (c) and (d) constitutes at least 10% by weight of the active detergent mixture, and the total of components (c) and (d) constitutes at least 25% by weight of the active detergent mixture.

10 14. A composition according to any one of the preceding claims wherein the weight of hydrotrope in the composition is not more than 12% calculated on the weight of the active detergent mixture.

15 15. A composition according to any one of the preceding claims which is an aqueous liquid composition containing 5 to 40% by weight of the active detergent mixture.

20 16. A detergent composition according to any one of the preceding claims wherein the alkyl ether sulphate (b) is provided by a mixture of materials of the general formula:

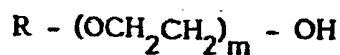


25

wherein R is a C₁₀ to C₁₈ alkyl group, X is a solubilising cation, and n, the average degree of ethoxylation, is from 2 to 12.

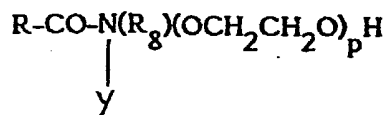
- 5 17. A detergent composition according to claim 16 wherein the alkyl ether sulphate (b) is provided by a primary alkyl ether sulphate in which the alkyl groups R are such that less than 20% of material of alkyl chain length C₁₄ and above is present, and the average degree of ethoxylation n is from 3 to 8.
- 10 18 A detergent composition according to any one of the preceding claims wherein both monoalkanolamide and dialkanolamide are present, in a weight ratio of monoalkanolamide to dialkanolamide in the range 2:1 to 1:2.
- 15 19. A detergent composition according to any one of the preceding claims wherein the non-ionic component (d) is at least one polyalkoxylated material.
- 20 20. A detergent composition according to claim 19 wherein the non-ionic component (d) is polyethoxylated and has an HLB value in the range from 12.0 to 16.0.
- 25 21. A detergent composition according to any one of the preceding claims wherein the non-ionic component (d) is selected from a polyethoxylated aliphatic alcohol of the general formula:

42.



wherein R is straight or branched alkyl having from 8 to 18 carbon atoms, and the average degree of ethoxylation \underline{m} is from 5 to 14, and/or

5 a polyethoxylated alkanolamide of the general formula



10 wherein R is a straight or branched alkyl having from 7 to 18 carbon atoms,

R_8 is an ethyleneoxy or propyleneoxy group

Y is hydrogen or $-\text{R}_8(\text{CH}_2\text{CH}_2\text{O})_q\text{H}$

p is 1 or more and q is 0, 1 or more.

15 22. A composition according to claim 21 wherein R has from 9 to 14 carbon atoms.

23. A composition according to claim 21 or claim 22 wherein the non-ionic component (d) is substantially only said polyethoxylated
20 aliphatic alcohol.

24. A method of preparing a liquid detergent composition which comprises mixing together materials as set forth in any one of the preceding claims and water, so as to form a composition as set forth in
25 any one of the preceding claims.

43.

25. A method of washing articles which comprises diluting a composition according to any one of claims 1 to 23 with water, and washing articles by hand in the resulting solution.